

AMMRC TR 70-36

AD

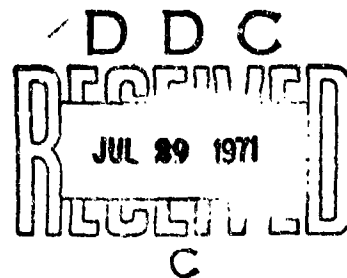
# WEAR- AND EROSION-RESISTANT COATINGS FOR TITANIUM ALLOYS IN ARMY AIRCRAFT

AD 726954

MILTON LEVY and JOSEPH L. MORROSSI  
METALS DIVISION

December 1970

Approved for public release; distribution unlimited.



ARMY MATERIALS AND MECHANICS RESEARCH CENTER  
Watertown, Massachusetts 02172

Reproduced by  
NATIONAL TECHNICAL  
INFORMATION SERVICE  
Springfield, Va 22151

23

ACCESSION FOR	
CPSTY	WRITE SECTION <input checked="" type="checkbox"/>
BOB	DIFF SECTION <input type="checkbox"/>
UNANNOUNCED	<input type="checkbox"/>
JUSTIFICATION	
BY	
DISTRIBUTION/AVAILABILITY CODES	
DATE	AVAIL. AND/OR SPECIAL
A	

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

Mention of any trade names or manufacturers in this report shall not be construed as advertising nor as an official indorsement or approval of such products or companies by the United States Government.

#### DISPOSITION INSTRUCTIONS

Destroy this report when it is no longer needed.  
Do not return it to the originator.

AMMRC TR 70-36

**WEAR- AND EROSION-RESISTANT COATINGS FOR TITANIUM ALLOYS IN  
ARMY AIRCRAFT**

Technical Report by

*MILTON LEVY and JOSEPH L. MORROSSI*

December 1970

D/A Project 1T062105A328

AMCMS Code 502E.11.294

Metals Research for Army Materiel

Agency Accession Number DA OB4762

Approved for public release; distribution unlimited.

METALS DIVISION

ARMY MATERIALS AND MECHANICS RESEARCH CENTER

Watertown, Massachusetts 02172

ARMY MATERIALS AND MECHANICS RESEARCH CENTER

WEAR- AND EROSION-RESISTANT COATINGS FOR  
TITANIUM ALLOYS IN ARMY AIRCRAFT

ABSTRACT

Diffusion-bonded electroless nickel plate was investigated as a wear-resistant coating for titanium alloys 6Al-6V-2Sn and 8Al-1Mo-1V. Plate adhesion and diffusion zone structure were assessed by metallographic and X-ray diffraction techniques. Effects of the diffusion heat treatments on mechanical properties and wear characteristics of the titanium alloys were determined. The diffusion bonding produced a surface of nickel-rich intermetallics which significantly improved the wear resistance of the titanium alloys without any appreciable degradation of their structural integrity.

## CONTENTS

	Page
ABSTRACT	
INTRODUCTION . . . . .	1
MATERIALS . . . . .	2
EXPERIMENTAL PROCEDURE . . . . .	3
RESULTS AND DISCUSSION . . . . .	4
EFFECT OF DIFFUSION BONDING TREATMENT ON MECHANICAL PROPERTIES OF THE ALLOYS	9
EVALUATION OF WEAR CHARACTERISTICS . . . . .	9
SUMMARY . . . . .	13
CONCLUSIONS . . . . .	15
LITERATURE CITED . . . . .	16

## INTRODUCTION

It was recognized early in the Army titanium program that the surface characteristics of titanium had many intrinsic shortcomings. For example, titanium alloys were very susceptible to galling, whereby transfer of metal occurs from one surface to another under sliding contact. Under severe conditions seizure occurs, whereby metal parts become welded together.

Because of its high strength-to-weight ratio, titanium had a potential for use in gun barrel liners. However, severe limitations were foreseen because of the reactivity of titanium with oxygen and nitrogen at elevated temperatures.

Recently, several problems associated with the use or potential use of titanium alloys in Army materiel have arisen. Dust ingestion has reduced time-between-overhaul (TBO) on helicopter T53 jet engines operating in Viet Nam. This dust has been responsible for erosion which is particularly severe in the compressor section. Dust erosion within the compressor section destroys the aerodynamic profile of axial blades and vanes and wears radial blades of the impeller (Ti-6Al-4V) so thin as to promote fatigue failure. Titanium alloys are also candidate materials for advanced aircraft engines in the compressor section where light weight, high strength, and ductility up to 1200 F (high pressure end) are required. Similar dust erosion problems, aggravated by higher temperatures, are anticipated.

In addition, significant weight reductions in helicopters can be achieved by substituting titanium alloys for steel in transmission components. The poor bearing characteristics of titanium make the development of a composite coating/base alloy system necessary for the utilization of titanium gears.

Approaches to the solution of titanium surface problems involve improved or modified processes for carburizing, nitriding, siliciding, induction surface hardening, coatings and chemical treatments to produce conversion coatings. Our approach will be limited to coatings.

Electrodeposited coatings of chromium and nickel have been considered for obtaining a wear-resistant surface on titanium. Electroplating studies carried out at this Center<sup>1,2</sup> demonstrated that only a moderate degree of adhesion of nickel or chromium could be achieved on titanium alloy surfaces. The key to adherent plate probably lies in the removal or modification of the oxide film prior to plating. Recently, Piontelli<sup>3</sup> claimed to have plated adherent chromium to titanium alloys 6Al-4V and 5Al-2.5Sn. His process consisted of anodizing, cathodically reducing the anodic film to 0.1 micron thickness (this film prevents the titanium from oxidizing in air), and finally electrodepositing chromium. He has reportedly<sup>4</sup> experienced some defoliation of chromium at corners or edges in bend tests.

Electroless nickel has also been considered for applications where resistance to wear and abrasion was of prime importance. In this technique a nickel salt is reduced with sodium hypophosphite on the surface being coated. The electroless plate is usually 93 to 95% nickel, the remainder being largely phosphorous, probably as nickel phosphide. One of the greatest advantages of

the process is that the coating deposits uniformly over irregular shapes, crevices, blind holes, and recesses. It thus is ideal for applications requiring plating of roots and tips of threads; worms, gears, etc. The nickel deposit is hard, nonporous and has a low coefficient of friction. Adhesion of the plate is good on most metals. However, on titanium the adhesion is inadequate.

Levy and Romolo<sup>6</sup> improved the adhesion of electroless nickel plate to several older vintage titanium alloys (150A, 155A, 6Al-4V) by diffusion bonding treatments. The interdiffusion between the nickel and titanium produced a wear-resistant surface which, under specific test conditions, was comparable to steel (case-hardened to  $R_c$  60). Our work reported here extends this study to include the more recently available titanium alloys 6Al-6V-2Sn and 8Al-1Mo-1V.

## MATERIALS

*Titanium Alloys:* Test specimens for adhesion, wear, and mechanical property evaluation were machined from titanium alloys 8Al-1Mo-1V and 6Al-6V-2Sn. The alloys were purchased in the annealed condition.

Ti-8Al-1Mo-1V is a near-alpha alloy containing 10 volume percent beta which is distributed at the grain boundaries. The alloy has been mainly used where weldability and moderate strength at or below 1000 F are required. It has potential for jet engine compressor components where superior resistance to creep and good tensile properties at temperatures between 750 and 1000 F are required. The alloy was used in the as-received annealed condition and had the following room temperature mechanical properties: yield strength 120,000 psi; ultimate tensile strength 133,000 psi; elongation 20%; and reduction of area 49%. The chemical analysis was: C 0.022%, Fe 0.06%, N 0.008%, Al 7.6%, V 1.1%, Mo 1.1%, H 0.005%, and O 0.09%.

Ti-6Al-6V-2Sn is an advanced alpha-beta composition which can be heat treated to very high strength levels. The alloy provides effective weight savings when extruded shapes, thick plate sections, or forged parts are required for air frame assemblies operating for long times up to temperatures of 700 F. It contains C 0.014%, Fe 0.79%, N 0.014%, Al 5.6%, V 5.7%, H 0.007%, Sn 2.0%, Cu 0.74%, O 0.14% and has the following room temperature mechanical properties in the as-received annealed condition: yield strength 137,000 psi; ultimate tensile strength 146,000 psi; elongation 20.5%; reduction of area 48.2%. For our study the Ti-6Al-6V-2Sn was heat treated to 160, 170, and 180 ksi yield strength levels. However, because of the similarities in results, only the data for the 160 ksi strength level alloy will be presented. Both alloys were processed for electroless nickel deposition and diffusion bonding heat treatments.

*Plating Bath:* The bath selected was a modified Brenner - Riddell<sup>7,8</sup> composition (see Table I<sup>9</sup>). All chemicals used were of reagent grade. The nickel chloride, ammonium chloride, sodium chloride, and sodium citrate were dissolved in distilled water, filtered, and the resultant solution heated. When the temperature reached 170 F, sodium hypophosphite previously dissolved in distilled water and filtered was added and the bath raised to 190 F. Aeration was accomplished with argon. The pH readings were taken every 15 minutes and the bath

adjusted to a pH of 8 to 9 by the addition of a 1:1  $\text{NH}_4\text{OH}$  solution. Replenishing solutions were added every 45 minutes: 250 cc of an aqueous solution containing 1.5 grams of nickel chloride per square decimeter of titanium surface area. An equal volume of aqueous solution containing 2 grams of sodium hypophosphite per square decimeter of surface area was also added.

#### EXPERIMENTAL PROCEDURE

The preparation of the test specimens involved the following sequence of operations: vapor degreasing, vapor blasting, activation, plating, aging, and heat treatment.

Prior to plating the specimens were trichloroethylene vapor degreased and vapor blasted with 100-grit glass beads at 70 to 80 psi. The specimens were immediately immersed in a slurry of glass beads for not more than 10 minutes, removed and placed in a stream of cold running water, and brushed until all grit was removed. The cleaned specimens were immediately immersed in an activating solution of hot (150 F), slightly acidic, 10% nickel chloride solution for 2 minutes and transferred into the plating bath. If plating did not start immediately, an aluminum rod was used to touch the samples, thus creating an internal galvanic cell, which initiated the reaction.

The plating rate was determined from Figure 1. After plating 0.5 to 0.6 mils of nickel, the specimens were rinsed in a stream of hot running tap water, air dried, and stored in a desiccator for at least 24 hours prior to heat treatment. The plated specimens were vacuum diffusion bonded at  $5 \times 10^{-7}$  Torr at temperatures between 750 and 1550 F for 4 hours and furnace cooled in vacuum.

Table I. BATH COMPOSITION AND PLATING CONDITIONS

Chemical	Formula	Liter	
		Grams	Moles
Nickel Chloride	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	50	0.126
Ammonium Chloride	$\text{NH}_4\text{Cl}$	50	.954
Sodium Chloride	$\text{NaCl}$	5	.085
Sodium Citrate	$\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$	100	.540
Sodium Hypophosphite	$\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$	10	.094

pH range: 8 to 9      Temp:  $190 \pm 2^\circ\text{F}$   
Agitation: constant with argon gas

$$\text{Molar Ratio} = \frac{\text{Ni}}{\text{Hypophosphite}} = \frac{0.126}{0.094} = 1.34$$

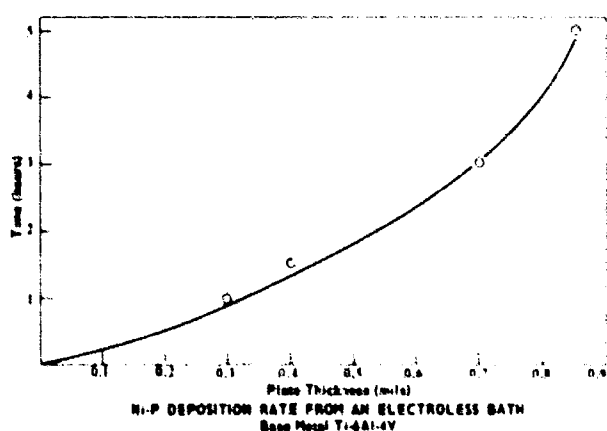


Figure 1. Ni-P DEPOSITION RATE FROM AN ELECTROLESS BATH. Base Metal Ti-6Al-4V



Plate adhesion and diffusion zone structure were assessed by metallographic and X-ray diffraction techniques. The effects of diffusion bonding on the mechanical properties of the alloys were determined. Wear resistance of the electroless nickel deposits, both diffused and undiffused, were obtained with a modified MacMillan apparatus.

## RESULTS AND DISCUSSION

*Metallographic Examination of Bond and Diffusion Zone Structure:* Figure 2 shows the microstructure of a cross section of electroless nickel plate on Ti-8Al-1Mo-1V in the as-plated condition, heat treated at 750, 950, 1150 and 1350 F for diffusion bonding. The phases present at the various levels of penetration are identified in Figure 3 (X-ray diffraction analysis after selective removal of material in 0.2 mil increments). In the as-plated condition adhesion is relatively poor. Adhesion is improved with increasing temperature and diffusion bonding is achieved at temperatures as low as 750 F. At 1350 F and above, several interdiffusion zones have been formed. Below 1350 F the main constituents of the outermost layer are Ni, Ni<sub>3</sub>Ti, and NiTi<sub>2</sub>. Above this temperature the nickel disappears and only the intermetallics are present. Figures 4 and 5 show that similar results were obtained for Ti-6Al-6V-2Sn with one exception. Nickel remains a constituent of the outer layer at 1450 F and disappears at 1550 F. For both titanium alloys the predominant phases present in the outermost layer and up to 0.5 mil into the diffusion zone are Ni and Ni<sub>3</sub>Ti (see Table II). Table III presents diffusion zone depths of the electroless nickel-titanium alloy combinations at the various temperature treatments. The diffusion zone depth increases with increasing temperature and is approximately the same for both alloys. A maximum penetration of 1.4 mils is noted.

NOT REPRODUCIBLE

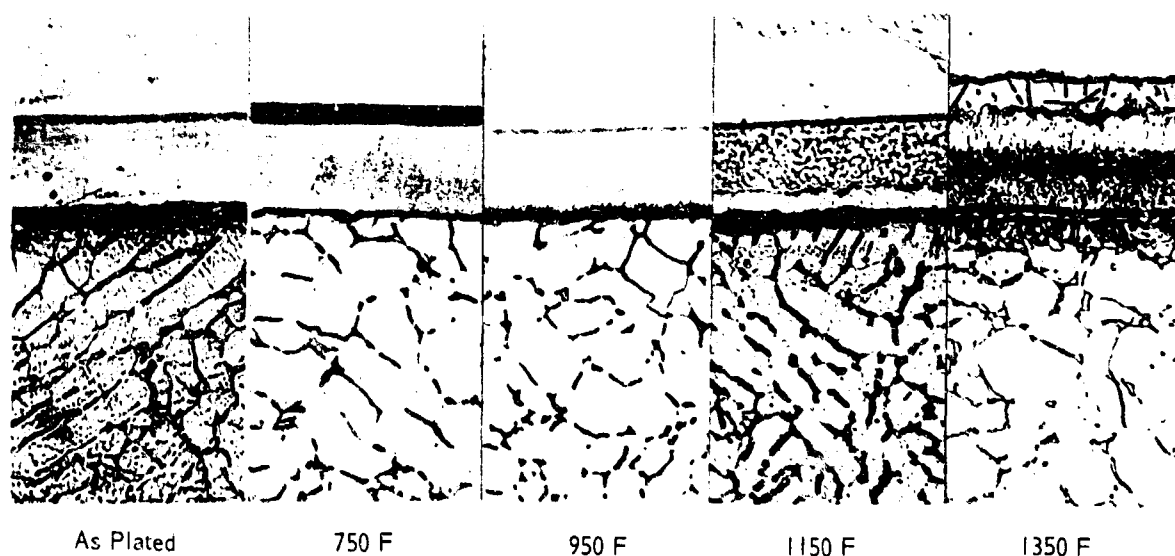


Figure 2. EFFECT OF HEAT TREATMENT ON ADHESION OF ELECTROLESS NICKEL TO Ti-8Al-1Mo-1V. Mag. 85X  
19-066-467/AMC-68

Table II. X-RAY DIFFRACTION ANALYSIS OF DIFFUSION  
BONDING OF ELECTROLESS NICKEL ON TITANIUM ALLOYS

Ti Alloys	Depth (mils)	750 F				950 F				1150 F				1350 F				1450 F				1550 F			
		Ni	Ni <sub>3</sub> Ti	NiTi <sub>2</sub>	Ti	Ni	Ni <sub>3</sub> Ti	NiTi <sub>2</sub>	Ti	Ni	Ni <sub>3</sub> Ti	NiTi <sub>2</sub>	Ti	Ni	Ni <sub>3</sub> Ti	NiTi <sub>2</sub>	Ti	Ni	Ni <sub>3</sub> Ti	NiTi <sub>2</sub>	Ti	Ni	Ni <sub>3</sub> Ti	NiTi <sub>2</sub>	Ti
8Al-1Mo-1V	0.0	A	B	C	-	A	B	C	-	A	B	C	-	-	A	C	-	-	A	C	-	-	A	D	-
	0.2	A	B	C	D	A	B	C	-	A	B	C	D	-	A	C	-	-	A	C	-	-	A	D	-
	0.4	A	B	C	C	A	B	C	C	A	B	C	D	-	A	C	-	-	A	C	-	-	A	B	-
	0.5													-	A	C	-								
	0.6	B	C	C	B					B	B	B	D	-	A	C	-	-	A	C	D	-	A	B	-
	0.7					D	-	-	B					-	A	C	C	-	A	B	C	-	B	B	C
	0.8	-	-	-	A	-	-	-	A	-	B	D	C	-	A	B	C	-	B	B	B	-	C	A	C
	0.9													-	B	B	C					-	-	A	B
	1.0					-	-	-	A	-	-	D	A	-	C	B	B	-	C	B	B	-	-	B	B
	1.1													-	-	-	A								
	1.2					-	-	-	A	-	-	-	A					-	-	D	A	-	-	C	A
	1.4																					-	-	C	A
6Al-6V-2Sn 160 KSI Y.S.	0.0	A	B	C	-	A	B	C	-	A	B	C	-	A	B	B	-	D	A	B	-	-	A	D	-
	0.2	A	B	C	-	A	B	C	C	A	B	C	D	A	B	B	-	-	A	B	-	-	A	C	-
	0.4	A	B	C	C	A	B	C	C	A	B	C	C	A	B	B	-	-	A	C	-	-	A	C	-
	0.6	A	B	C	B	B	B	C	B	A	B	C	C	A	A	C	D	-	A	B	D				
	0.8	-	-	-	A	D	-	-	A	A	B	C	B	A	B	C	C	-	A	C	C	-	B	B	C
	1.0									-	-	-	A	C	C	C	B	-	B	C	B	-	D	B	B
	1.2													-	-	-	A	-	C	D	A	-	-	C	A
	1.4																	-	-	-	A	-	-	-	A
170 KSI Y.S.	0.0	A	B	C	-	A	B	B	-	A	B	C	-	A	B	B	-	D	A	B	-	-	A	D	-
	0.2	A	B	C	-	A	B	C	-	A	B	B	D	A	B	B	-	-	A	B	-	-	A	D	-
	0.4	A	B	C	C	A	B	C	D	A	B	B	D	A	B	B	-	-	A	C	-	-	A	D	-
	0.6	A	B	C	B	A	B	C	C	B	B	B	C	A	B	B	D	-	A	C	-	-	A	D	-
	0.8	-	-	-	A	-	-	-	A	C	C	C	B	C	B	B	D	-	A	D	-	-	A	C	D
	1.0									-	-	-	A	D	B	B	D	-	B	D	C	-	A	B	C
	1.2													-	C	C	B	-	D	-	A	-	B	B	B
	1.4													-	-	D	A	-	-	-	A	-	-	C	A
180 KSI Y.S.	0.0	A	B	C	-	A	B	C	-	A	B	B	-	A	B	B	-	D	A	B	-	-	A	D	-
	0.2	A	B	C	C	A	B	B	C	A	B	B	-	A	B	B	-	-	A	B	-	-	A	C	-
	0.4	A	C	C	B	A	B	B	B	A	B	B	D	A	B	B	-	-	A	C	-	-	A	C	-
	0.6	C	D	D	A	-	-	-	A	A	B	B	D	B	B	B	D	-	A	C	D	-	A	C	-
	0.8	-	-	-	A	-	-	-	A	-	-	-	A	C	B	B	C	-	A	C	C	-	A	B	D
	1.0													-	C	B	B	-	B	D	C	-	A	B	D
	1.2													-	D	C	A	-	D	D	A	-	B	B	C
	1.4													-	-	-	A	-	-	-	A	-	-	C	A

A Predominant

B Moderate

C Slight

D Trace

NOT REPRODUCIBLE

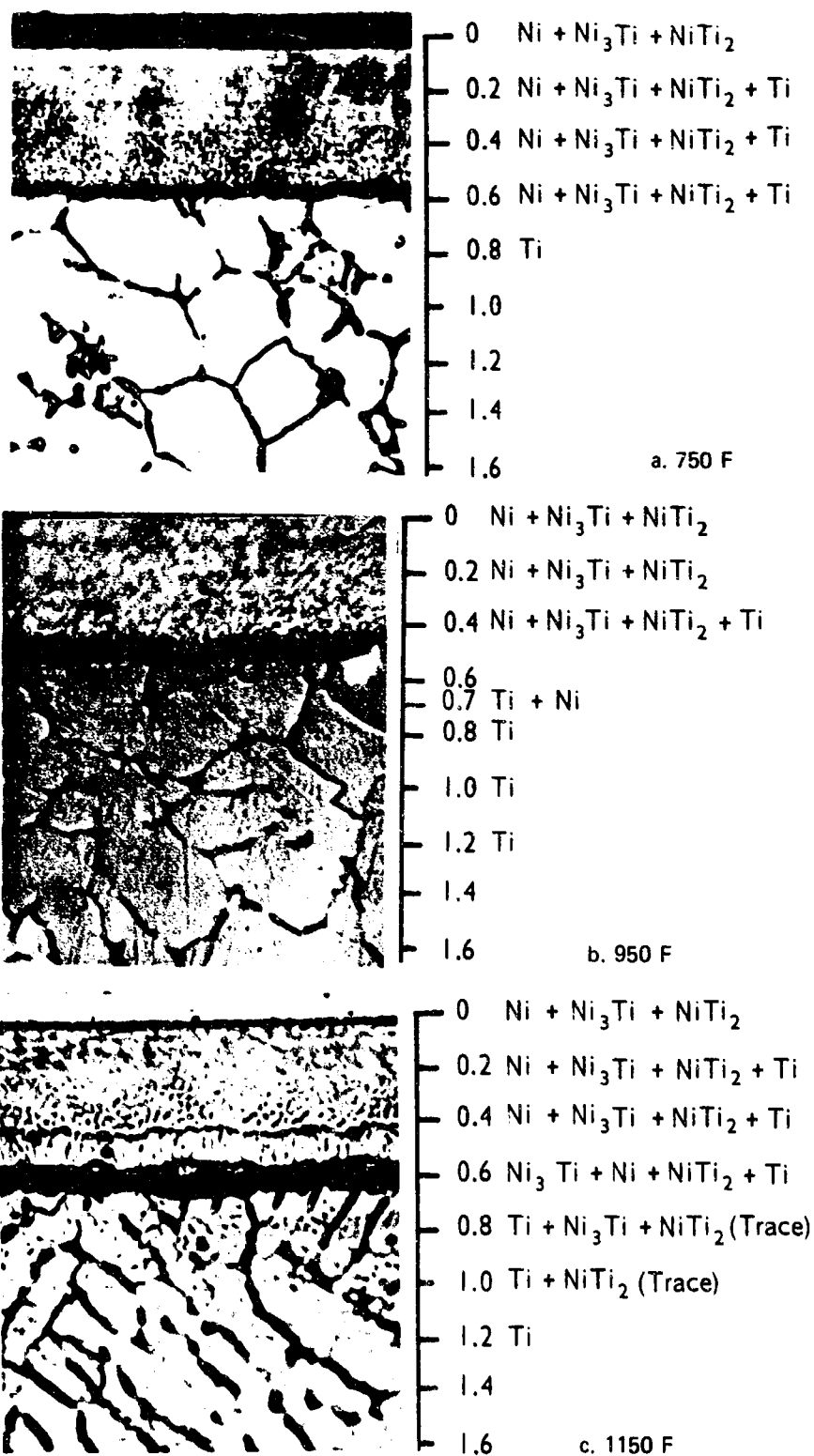
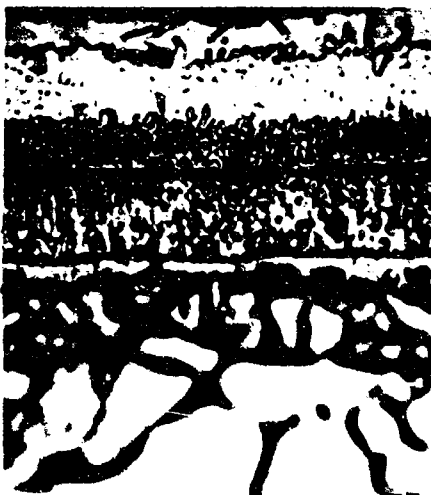


Figure 3. ELECTROLESS NICKEL DIFFUSION BONDED TO Ti-8Al-1Mo-1V AT VARIOUS TEMPERATURES .  
DIFFUSION ZONE STRUCTURE. Mag. 1500X

NOT REPRODUCIBLE



- 0  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.2  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.4  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.6  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.7  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 + \text{Ti}$
  - 0.8  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 + \text{Ti}$
  - 0.9  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 + \text{Ti}$
  - 1.0  $\text{NiTi}_2 + \text{Ni}_3\text{Ti} + \text{Ti}$
  - 1.1  $\text{Ti}$
  - 1.2
  - 1.4
  - 1.6
- d. 1350 F



- 0  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.2  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.4  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.6  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 + \text{Ti (Trace)}$
  - 0.8  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 + \text{Ti}$
  - 1.0  $\text{NiTi}_2 + \text{Ni}_3\text{Ti} + \text{Ti}$
  - 1.2  $\text{Ti} + \text{NiTi}_2$
  - 1.4
- e. 1450 F



- 0  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 \text{ (Trace)}$
  - 0.2  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 \text{ (Trace)}$
  - 0.4  $\text{Ni}_3\text{Ti} + \text{NiTi}_2 \text{ (Trace)}$
  - 0.6  $\text{Ni}_3\text{Ti} + \text{NiTi}_2$
  - 0.8  $\text{NiTi}_2 + \text{Ni}_3\text{Ti (Trace)}$
  - 1.0  $\text{Ti} + \text{NiTi}_2$
  - 1.2  $\text{Ti} + \text{NiTi}_2$
  - 1.4  $\text{Ti} + \text{NiTi}_2$
  - 1.6
- f. 1550 F

Figure 3. ELECTROLESS NICKEL DIFFUSION BONDED TO Ti-8Al-1Mo-1V AT VARIOUS TEMPERATURES  
DIFFUSION ZONE STRUCTURE. Mag. 1500X

NOT REPRODUCIBLE

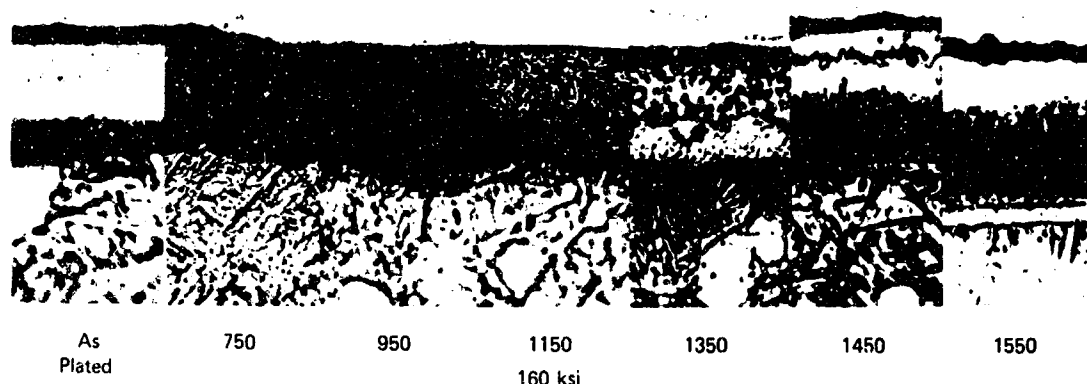


Figure 4. EFFECT OF HEAT TREATMENT ON ADHESION OF ELECTROLESS NICKEL TO Ti-8Al-1Mo-1V. Mag. 850X

Table III. DEPTH OF DIFFUSION BONDING OF ELECTROLESS NICKEL ON TITANIUM ALLOYS

Diffu- sion Temp, (deg F)	Depth, mils				
	Ti-6Al-6V-2Sn				Ti-8Al- 1Mo-1V
	160 ksi	170 ksi	180 ksi	Aver- age	
R.T.	0.6	0.6	0.6	0.6	0.6
750	0.6	0.6	0.6	0.6	0.6
950	0.6	0.6	0.6	0.6	0.6
1150	0.8	0.8	0.6	0.8	0.8
1350	1.0	1.2	1.2	1.2	1.0
1450	1.2	1.2	1.2	1.2	1.2
1550	1.2	1.4	1.4	1.4	1.4

Diffusion bonded 4 hours at temperature

X-ray diffraction analysis did not yield any traces of NiTi or NiP. Duwez and Taylor<sup>10</sup> reported that eutectoidal decomposition of TiNi occurs on prolonged heating at 1202 and 1472 F, the phases Ti<sub>2</sub>Ni and TiNi<sub>3</sub> being formed. Poole and Hume-Rothery<sup>11</sup> confirmed their conclusion. According to Purdy and Parr,<sup>12</sup> TiNi does not undergo a low-temperature eutectoid decomposition. However, the possible decomposition of TiNi could explain its absence in our X-ray diffraction tracings. Chemical analysis of our electroless nickel plate showed the phosphorous content to be 6% on Ti-8Al-1Mo-1V and 9% on the Ti-6Al-6V-2Sn. According to Gutzeit<sup>9</sup> the phosphorous content of electroless nickel plate varies between 3 and 16% depending on the bath and plating parameters. Graham et al.<sup>13</sup> believe that the deposit is a supersaturated solution of phosphorous in crystalline nickel. Randin et al.<sup>14</sup> confirm this and report that the X-ray diffraction lines of nickel and nickel phosphide coincide wholly or in part and since the grain size of the as-plated electroless nickel is extremely small, broadening of the diffracted lines occurs. Thus the phosphide phase would be hardly discernible.

# EFFECT OF DIFFUSION BONDING TREATMENT ON MECHANICAL PROPERTIES OF THE ALLOYS

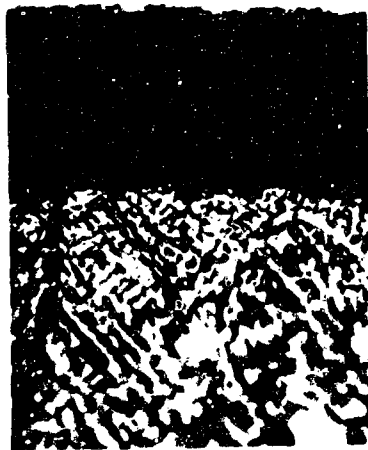
Table IV gives the microhardness and converted Rockwell C hardness values for the titanium alloys before and after the electroless nickel plating and subsequent diffusion bonding treatment. In the unplated condition, the higher strength Ti-6Al-6V-2Sn alloy was markedly harder than Ti-8Al-1Mo-1V, which was expected. Significant increases in hardness were achieved for both alloys by the plating and subsequent diffusion bonding treatments at the two extremes of temperature, 750 and 1550 F ( $R_C$  57 for Ti-6Al-6V-2Sn and  $R_C$  50 for Ti-8Al-1Mo-1V). The effects of the plating and diffusion bonding on the tensile strength, yield strength, impact energy, elongation and reduction of area of the alloys are shown in Table V. The nickel plating had little or no effect on the mechanical properties of both alloys. For Ti-8Al-1Mo-1V the diffusion bonding treatments (between 750 and 1350 F) had no degradative effects on room temperature tensile and yield strengths. There was an approximately 10% reduction in elongation and reduction of area while Charpy impact resistance was enhanced. Where diffusion temperatures exceed the aging temperature for the alloy, some degradation in mechanical properties can be expected. For the Ti-6Al-6V-2Sn tensile and yield strengths were unaffected by heat treatments between 750 and 1150 F. The heat treatment at 1350 F caused approximately a 5% reduction in these properties. Again Charpy impact resistance increased, but a significant increase rather than decrease occurred in elongation and reduction of area at 1350 F.

## EVALUATION OF WEAR CHARACTERISTICS

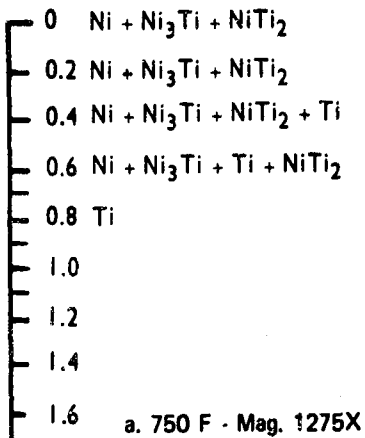
The wear resistance of the titanium alloys, the electroless nickel deposits both diffused and untreated, were determined with a modified MacMillan Wear Tester which has been described in detail elsewhere.<sup>6</sup> Briefly, it uses the outer race of a tapered bearing as a specimen which rotates against the full width of a

Table IV. MICROHARDNESS OF DIFFUSION BONDING  
OF ELECTROLESS NICKEL ON TITANIUM ALLOYS

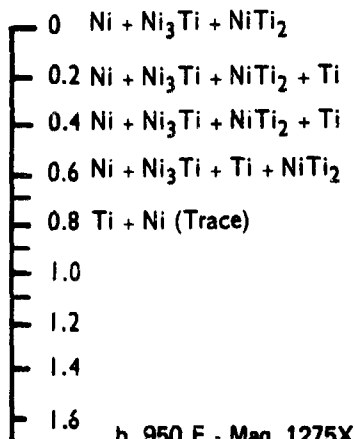
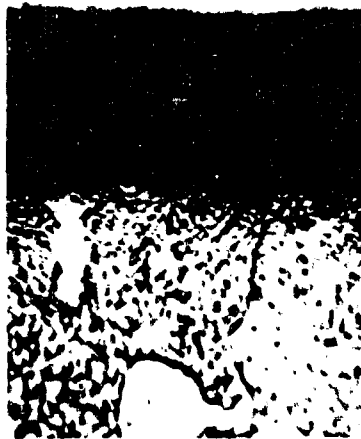
Diffu- sion Temp, (deg F)	Hardness									
	Ti-6Al-6V-2Sn								Ti-8Al-1Mo-1V	
	160 ksi	170 ksi	180 ksi	160 ksi	170 ksi	180 ksi	Average			
	Knoop			R <sub>C</sub> (conv)			Knoop	R <sub>C</sub>		
Not plated	507	518	563	47.8	48.5	51.3	-	-	367	37.0
R.T.	535	539	526	49.6	49.8	49.0	533	49.5	538	49.8
750	668	651	718	56.9	56.0	59.4	679	57.4	534	50.0
950	550	548	572	50.5	50.4	52.0	557	51.0	415	41.0
1150	433	448	465	43.0	42.9	45.0	449	43.6	392	39.0
1350	378	398	380	37.7	39.6	38.0	385	38.4	452	46.0
1450	549	509	537	50.4	48.0	49.7	532	49.4	-	-
1550	684	688	684	57.7	58.0	57.1	685	57.6	-	-



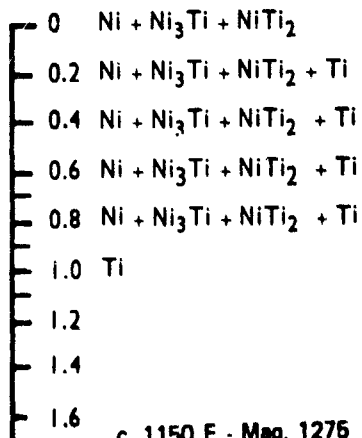
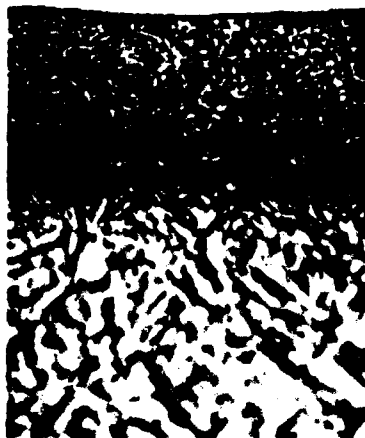
NOT REPRODUCIBLE



a. 750 F - Mag. 1275X



b. 950 F - Mag. 1275X



c. 1150 F - Mag. 1275

Figure 5. ELECTROLESS NICKEL DIFFUSION BONDED TO Ti-6Al-8V-2Sn AT VARIOUS TEMPERATURES. DIFFUSION ZONE STRUCTURE, 160 KSI YIELD STRENGTH

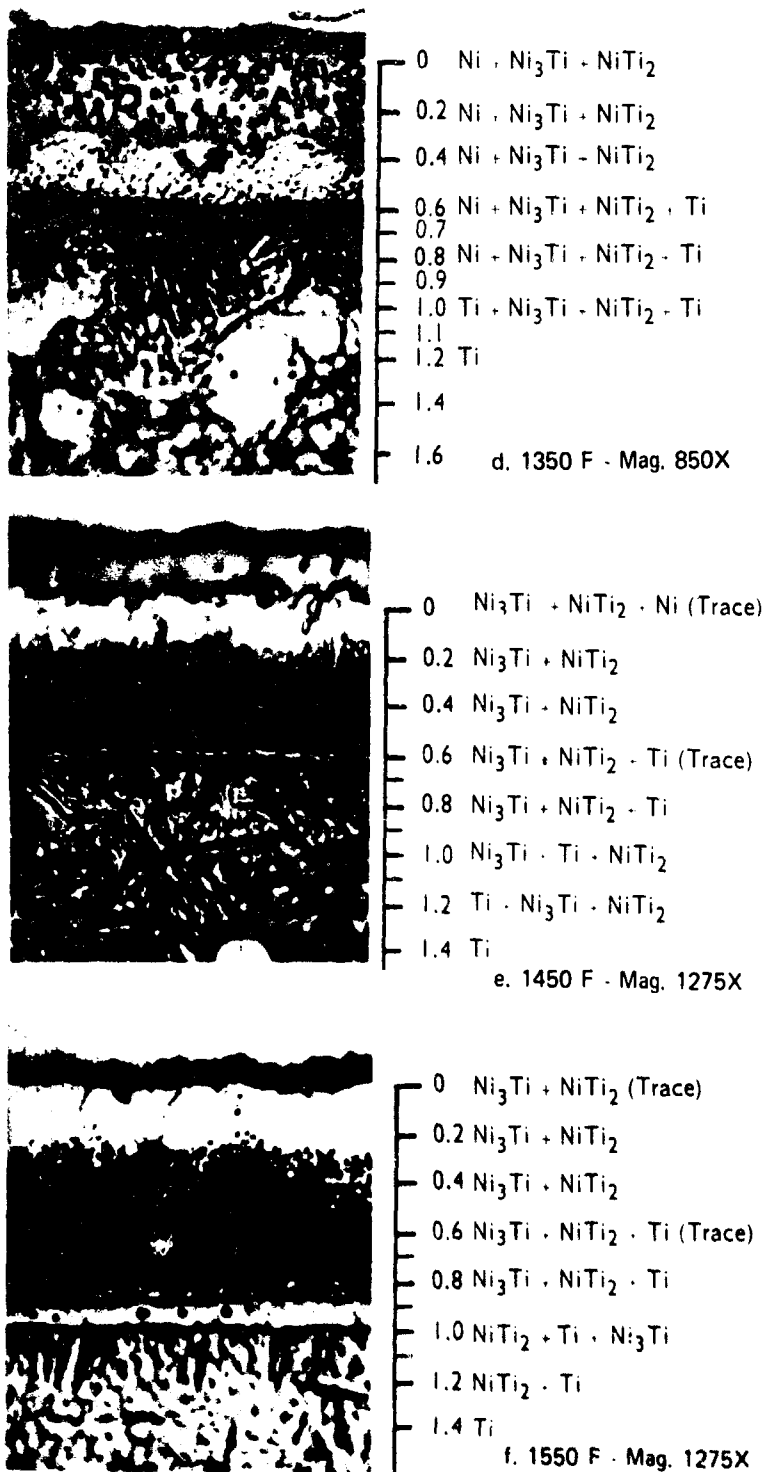


Figure 5. ELECTROLESS NICKEL DIFFUSION BONDED TO Ti-6Al-6V-2Sn AT VARIOUS TEMPERATURES  
 DIFFUSION ZONE STRUCTURE, 160 KSI YIELD STRENGTH



Table V. MECHANICAL PROPERTIES OF TITANIUM ALLOYS  
ELECTROLESS NICKEL PLATED AND DIFFUSION BONDED

Diffusion Temperature (deg F)	Ti-8Al-1Mo-1V		Ti-6Al-6V-2Sn					
			160 ksi		170 ksi		180 ksi	
	Plated	Not Plated	Plated	Not Plated	Plated	Not Plated	Plated	Not Plated
a. Tensile Strength (ksi)								
Room Temp	-	137.0	166.8	169.6	172.1	171.8	182.3	182.7
750	137.4	136.9	171.5	169.6	173.0	170.8	187.9	187.3
950	137.1	137.1	169.4	170.0	171.4	170.9	175.8	183.4
1150	138.8	135.3	168.6	167.1	169.5	168.6	180.3	178.5
1350	135.3	136.0	159.0	159.4	158.0	153.4	158.0	156.4
b. 0.2% Yield Strength (ksi)								
Room Temp	-	127.5	162.3	160.9	168.5	167.5	179.3	179.3
750	124.8	127.3	165.5	161.8	169.0	167.8	184.0	182.5
950	129.8	137.1	165.0	163.2	167.5	167.5	175.5	175.3
1150	131.3	128.3	164.5	161.0	163.9	165.8	176.8	175.0
1350	127.0	125.8	155.0	152.5	152.0	151.5	153.0	153.0
c. Impact Energy (ft-lb)								
Room Temp	18.9	17.8	10.2	11.1	9.2	9.2	8.1	8.3
750	17.0	18.3	10.3	10.1	8.5	9.6	8.0	8.0
950	17.7	20.0	8.7	10.0	7.7	9.1	6.4	7.8
1150	19.1	20.0	9.3	12.9	8.3	12.3	7.6	8.8
1350	20.1	24.5	13.0	15.5	10.9	11.5	11.5	14.0
d. Elongation (%)								
Room Temp	-	17.9	12.5	12.2	11.5	10.0	10.0	10.7
750	14.7	17.2	10.4	11.8	9.3	12.2	8.6	7.1
950	15.7	19.3	7.1	9.3	7.9	10.0	5.7	6.8
1150	17.1	17.5	14.3	14.3	10.7	12.2	7.2	8.2
1350	14.7	17.9	17.9	16.4	15.0	17.9	13.3	16.4
e. Reduction of Area (%)								
Room Temp	-	40.5	29.5	27.6	17.1	23.3	31.5	34.0
750	33.8	34.7	18.9	30.5	22.5	34.2	21.6	20.1
950	34.3	37.4	10.3	16.9	18.2	24.1	10.9	21.3
1150	35.2	33.8	35.3	36.5	30.2	33.2	30.3	32.2
1350	28.5	38.9	38.9	42.8	37.6	52.1	29.8	48.2

stationary block under load. Failure occurs when either the preset torque of 11 to 13 ft-lb is exceeded or a rise in temperature of 100 F above ambient occurs. The wear data obtained are compared with 52100 steel (case hardened to  $R_c$  60) in Table VI. Both titanium alloys in the unplated and as-plated conditions fail in 6 minutes or less. Photographs of the failed specimens contained in Figure 6 show that the as-plated material fails after defoliation of plate occurs, attesting to the poor bond in the as-plated condition. The diffusion bonding treatments markedly improve wear resistance which increases with increasing temperatures. Wear resistance is almost comparable to steel at the 750 F heat treatment and significantly better than steel under the same conditions at the higher temperature treatments.

### SUMMARY

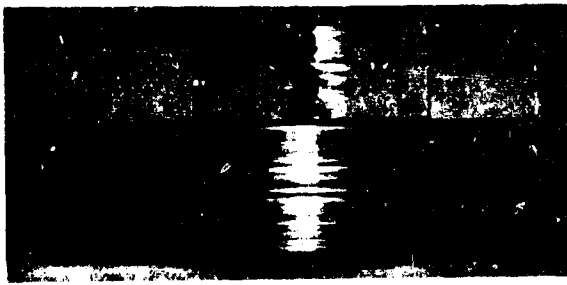
Electroless nickel in the as-plated condition is poorly bonded to titanium alloys. The diffusion treatments between 750 and 1550 F significantly improve adhesion. For the Ti-8Al-1Mo-1V, the diffusion treatments between 750 and 1150 F produce Ni,  $Ni_3Ti$ , and  $NiTi_2$  (in order of decreasing amounts) in the outer layer; above this temperature (1350 to 1550 F),  $Ni_3Ti$  and  $NiTi_2$ . For the Ti-6Al-6V-2Sn alloy, Ni,  $Ni_3Ti$ , and  $NiTi_2$  are formed between 750 and 1350 F, while at 1450 to 1550 F  $Ni_3Ti$  and  $NiTi_2$  only are present. We see that a higher temperature is required to completely transform the nickel into the intermetallics for the 6Al-6V-2Sn alloy (1550 F versus 1350 F).

The diffusion treatments between 750 and 1350 F did not degrade the mechanical properties of the Ti-8Al-1Mo-1V alloy. There was a 5% reduction in ultimate tensile strength and yield strength of the Ti-6Al-6V-2Sn alloy at 1350 F. Wear data show a marked improvement attributed to the electroless nickel plate and diffusion treatments. Best results were obtained at the highest diffusion temperatures, 1450 and 1550 F where the major constituents of the outer surface are

Table VI. EFFECT OF DIFFUSION BONDING OF ELECTROLESS NICKEL ON TITANIUM ALLOYS ON WEAR RESISTANCE USING THE MODIFIED MacMILLAN WEAR TESTER

Diffusion Temperature (deg F)	Time for Failure, Minutes				
	Steel on Steel 52100 $R_c$ 60	Ti-8Al-1Mo-1V	Ti-6Al-6V-2Sn		
			160 ksi	170 ksi	180 ksi
Not plated	136	6.0	5.3	-	-
Room temp. as plated	-	6.0	5.0	5.1	5.7
750	-	91	115	128	124
950	-	202	210	210	200
1150	-	334	252	302	296
1350	-	574	326	371	347

NOT REPRODUCIBLE



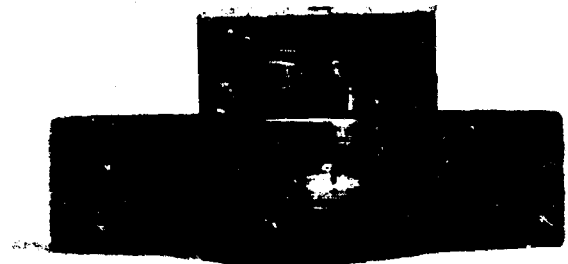
Not Plated  
Failure, 6 Minutes



Diffusion Bonded at 950 F  
Failure, 202 Minutes



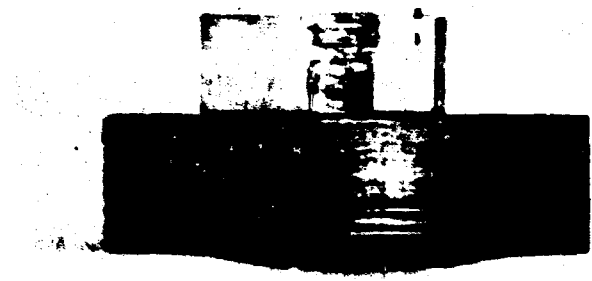
As Plated  
Failure, 6 Minutes



Diffusion Bonded at 1150 F  
Failure, 334 Minutes



Diffusion Bonded at 750 F  
Failure, 91 Minutes



Diffusion Bonded at 1350 F  
Failure, 574 Minutes

Figure 6 MODIFIED MACMILLAN WEAR TEST OF DIFFUSION BONDED  
ELECTROLESS NICKEL ON Ti-8Al-1Mo-1V Mag. 2X

the intermetallics  $\text{Ni}_3\text{Ti}$  and  $\text{NiTi}_2$ . The best wear characteristics are therefore obtained at diffusion temperatures which degrade the mechanical properties of the alloys by about 5%. One therefore has to make a compromise in selecting the optimum diffusion treatment, that is, the temperature at which the best wear is obtained without any degradation of mechanical properties. On this basis 1350 F is recommended for Ti-8Al-1Mo-1V and 1150 F for Ti-6Al-6V-2Sn. At these diffusion temperatures the wear resistance of the coated alloys is still far superior to the wear resistance of case-hardened steel under the conditions of test. In general, the best compromise will most likely depend on the prior heat treatment of the alloys.

## CONCLUSIONS

We have demonstrated that the diffusion bonding of electroless nickel plate is a promising technique for producing a wear-resistant surface for titanium alloys. However, at this point we cannot infer that this development will solve important Army problems associated with titanium compressor components of gas turbine engines where erosion resistance is of prime importance, or transmission gears in the drive system of helicopters where contact stresses and sliding velocities are all-important. For such applications further testing is necessary whereby the conditions of the test better simulate actual service conditions. Accordingly, in the near future we plan to carry out the following testing programs:

1. erosion testing utilizing a system which is capable of multi-angular abrasive particle impingement at temperatures ranging from ambient to 800 F, with controlled air and particle velocities - simulates dust ingestion in Viet Nam operations;
2. wear testing whereby the combined effects of contact stresses and sliding velocities will be exerted on the coated alloy system in a geared roller tester; and
3. for both applications, fatigue testing utilizing rotating beam specimens.

Since the diffusion bonding treatments have provided a metallurgically bonded nickel-rich surface on titanium, the feasibility of depositing adherent electroplated chromium over titanium alloys is greatly enhanced. Indeed, initial experiments have shown that adherent chromium plate can be deposited over titanium alloys which have been electroless nickel plated and diffusion bonded. Further evaluation for adhesion, wear, and erosion resistance is highly recommended and planned.

## LITERATURE CITED

1. LEVY, C. *Electroplating on Titanium*. WAL TR 401/1, July 1952.
2. LEVY, C. *Chromium Plating on Titanium Alloys*. Metal Finishing, May 1960.
3. PIONTELLI, R. Politecnico Istituto, Milan, Italy.
4. SULLIVAN, T. E., and MARKUS, H. Report of Visit to Politecnico Istituto, Milan, Italy.
5. BRENNER, A., and RIDDELL, G. E. *Nickel Plating on Steel by Chemical Reduction*. Proc. Am. Electroplaters Soc., v. 33, 1946, p. 16.
6. LEVY, M., and ROMOLO, J. *Improved Adhesion of Electroless Nickel Plate on Titanium Alloys*, 48th Annual Technical Proceedings Am. Electroplaters Soc., 1961, p. 135-141.
7. U. S. A. Patent No. 2,253,283, December 5, 1950.
8. ASTM Special Publication No. 265, Symposium on Electroless Nickel Plating, Am. Soc. Test. Mat., 1959.
9. GUTZEIT, G. *Plating*, v. 6, 1959, p. 1158.
10. DUWEZ, P., and TAYLOR, J. L. *The Structure of Intermediate Phases in Alloys of Titanium with Iron, Cobalt, and Nickel*. Trans. AIME, v. 188, 1950, p. 1173-1177.
11. POOLE, D. M., and HUME-ROTHERY, W. *The Equilibrium Diagram of the System Nickel-Titanium*. J. of the Institute of Metals, v. 83, 1954-55, p. 473-480.
12. PURDY, G. R., and PARR, J. G. Trans. AIME, v. 221, 1961, p. 636-639.
13. GRAHAM, A. H., LINDSAY, R. W., and READ, H. J. *The Structure and Mechanical Properties of Electroless Nickel*. J. Electrochem. Soc., v. 112, 1965, p. 401.
14. RAMDIN, J. P., MAIRE, P. A., SAUER E., and HINTERMANN, H. E. *Data and X-Ray Studies of Electroless Nickel*. J. Electrochem. Soc., v. 114, no. 5, 1967. p. 442.

## DOCUMENT CONTROL DATA - R &amp; D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Army Materials and Mechanics Research Center Watertown, Massachusetts 02172		2a. REPORT SECURITY CLASSIFICATION Unclassified	
		2b. GROUP	
3. REPORT TITLE WEAR- AND EROSION-RESISTANT COATINGS FOR TITANIUM ALLOYS IN ARMY AIRCRAFT			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (First name, middle initial, last name) Milton Levy and Joseph L. Morrossi			
6. REPORT DATE December 1970		7a. TOTAL NO. OF PAGES 19	7b. NO. OF REFS 14
8a. CONTRACT OR GRANT NO.		9a. ORIGINATOR'S REPORT NUMBER(S) AMMRC TR 70-36	
b. PROJECT NO. D/A 1TO62105A328			
c. AMCMS Code 502E.11.294		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
d. Agency Accession Number DA OB4762			
10. DISTRIBUTION STATEMENT Approved for public release; distribution unlimited.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY U. S. Army Materiel Command Washington, D. C. 20315	
13. ABSTRACT Diffusion-bonded electroless nickel plate was investigated as a wear-resistant coating for titanium alloys 6Al-6V-2Sn and 8Al-1Mo-1V. Plate adhesion and diffusion zone structure were assessed by metallographic and X-ray diffraction techniques. Effects of the diffusion heat treatments on mechanical properties and wear characteristics of the titanium alloys were determined. The diffusion bonding produced a surface of nickel-rich intermetallics which significantly improved the wear resistance of the titanium alloys without any appreciable degradation of their structural integrity. (Authors)			

DD FORM 1473  
1 NOV 66REPLACES DD FORM 1473, 1 JAN 64, WHICH IS  
OBSOLETE FOR ARMY USE.UNCLASSIFIED  
Security Classification

KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Titanium alloys Helicopters Electroless plating Diffusion coating Surfaces Adhesion Wear Erosion Mechanical properties Protective coatings						